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INFORMATION REPORT

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COUNTRY East Germany

REPORT

SUBJECT VEB Hydrierwerk Zeitz Research Projects during 1954 and the First Quarter of 1955.

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1. In accordance with the approved plan for research and technology, eleven research projects were undertaken in 1954 at the Research and Development Office of VEB Hydrierwerk Zeitz. The research projects were divided up among the individual departments at Zeitz as follows:

- a. Research and Development Office -- Six research projects.
- b. Procedure Technology Department -- Two research projects.
- c. High Pressure Department -- Two research projects.
- d. Measurements Department -- One research project.

The research was concerned with the following: Low-temperature hydrogenation; a separate light oil process (getrennte Leichtoelfahrweise) for the purpose of obtaining gasoline of a high octane rating and containing aromatics; the reduction of the high ash content in Espenhain tar; reactivity properties of hydrogenation catalytic agents; analysis of the components of "Einspritz" and "Abstreifer" products with the goal of developing a new industrial analysis process; the development of a petro-chemistry by obtaining individual hydro-carbons and by further chemically transforming them; and a new x-ray contrast agent for non-ferrous metals to be used in material tests. The subjects of the research projects were intimately connected with Zeitz's production and were to promote the further development of the low-temperature hydrogenation process and to make possible an increase in types of products.

2. In general, all of the research projects included in the plan were successfully carried out to a great extent. Of the eleven, five had been completed at the end of the year and complete final reports on them had been written. The remaining six projects were to be continued during 1955.

* Literally, injection and strip

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3. The approved plan funds for the eleven projects originally amounted to 1,450,000 DME. This amount was reduced to 1,305,000 DME as a result of decreases in funds approved by the Ministry for Heavy Industry. This difference of 145,000 DME was deducted from those projects which in the third quarter had used the least amount of funds. The original planned costs for the catalytic light oil processing research project called for 187,000 DME in production costs and 133,000 DME in basic funds (Grundmittel); after the preliminary project had been completed, however, basic funds were increased to 228,000 DME and production costs reduced to 92,000 DME. Further, 22,000 DME from the basic funds of the individual hydro-carbons projects and 18,000 DME from the basic funds of the thiophene project were transferred to the T-ester project, and finally 20,000 DME were taken from the basic funds of the T-ester project and applied to the planned 1955 funds for the same project. The planned funds for all research topics at Zeitz amounted to 1,285,000 DME at the end of plan year 1954. Actual costs, including the basic funds, amounted to 1,265,000 DME; that is, despite the 10% reduction of the original planned funds for 1954, the finance plan of the Research and Development Office at Zeitz was fulfilled.

4. The following is a short description of the individual research projects carried out by the Research and Development Office in 1954:

a. [redacted] Designation: Lubricating Oil Analysis

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(Schmieroelanalytik):

After a thorough study of available literature on the subject had been made and after the necessary apparatus had been procured, synthetic lubricating oils such as paraffin, crack olefine polymerisate, Voltol oils and other products of the hydrogenation of lignite tars, were analyzed. Physical and chemical analysis was carried out, using known methods, after the synthetic lubricating oils had been reduced to their basic components by distillation and chromatography. The various synthetic oils required various methods of analysis, from which the basic components could be determined and the quality of the end product controlled. Attempts to determine relationships between basic components and other properties were first completed for low-temperature hydrogenation machine oil and revealed new facts, especially concerning friction properties; thus it will be necessary to intensify this work. The project is to be continued in 1955 in an expanded form under the designation "Lubricating Oil--Refining and Analysis". The approved plan funds for the project amount to 120,000 DME. (See paragraph 7, a).

b. [redacted] Designation: Low-Temperature Hydrogenation Process (TH-Verfahren):

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Within the framework of this research project, technical experiments on a small scale consisting of varying low-temperature hydrogenation conditions were carried out, and knowledge was gained which will improve the technology of the large-scale technical process. The study of the influences of temperature, capacity, gas charge and pressure was carried out with old contacts and revealed a few facts which are important for the large-scale technical process, such as: the increase of the cracking reaction as the catalytic agent grows older; the increased sensitivity to variations of the amount of oil (Oeldurchsatz); the slight influence of H_2 partial pressure, which can be compensated for by increasing the temperature by 20° when pressure is reduced to 100 atmospheres; and the slight influence of the size of the contact granules. The project was completed at the end of the plan year 1954.

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c. Designation: Hydro-Carbon Analysis

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(Kw-Analytik):

A survey was made of all literature concerning modern methods for determining the components of hydro-carbon mixtures on a physical-chemical basis and current knowledge of lignite low-temperature tar and light oil. All "Abstreifer" products produced at VEB Hydrierwerk Zeitz, up to and beyond the gasoline range, as well as unhydrogenated and perhydrogenated light oil, were analyzed for chemical and physical properties, and quantitative assays of the basic components were made after the products have been reduced to their basic components by precision distillation and chromatography. It was found possible to reduce Espenhain tar to its basic components by pretreating it chemically and then subjecting it to precision vacuum distillation and chromatography and thus to determine by analysis the C-distribution and ring number of the hydro-carbon portion and to establish qualitatively the probable components of the tar. This project is to be continued in 1955 (see paragraph 7, b); approved plan funds amount to 130,000 DME.

d. Designation: Catalytic Light Oil Processing
(Katalytische Leichtöelverarbeitung):

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Experiments with the process developed by VEB Hydrierwerk Zeitz for the selective hydrogenation of previously dephenolized light oil for the purpose of obtaining gasoline with high octane ratings were continued in the small apparatus which was available. On the basis of results obtained from these experiments, a semi-technical installation with a capacity of 100 liters was built and put into operation. The results of the experiments were completely confirmed. The installation is currently being used chiefly for the study of the coking problem (Verkokungsproblem). The experiments carried out on a large technical scale in the dehydrogenation installation at Boehlen were so successful that the process was introduced on a large technical scale in Zeitz and by the end of 1954 had already become one of the basic parts of technology. The project was completed at the end of plan year 1954.

e. Designation: "T-ester":

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The process which had been worked out in the laboratory was put into operation on a small technical scale, and as a result of a series of extensive experiments, the optimal conditions for a technically feasible process were determined. The individual reaction stages were coordinated with each other for the purpose of attaining the greatest possible efficiency. A preliminary project for the establishment of a semi-technical installation was worked out, and construction and assembly of the installation was carried out to a great extent. In the laboratory, experiments on regenerating the oxidation agent and replacing the catalytic agent by cheaper products were begun. The project is to be continued through December 1955 (see paragraph 7, d). The approved plan funds amount to 300,000 DME.

f. Designation: Refining of Lubricating Oil
(Schmieröelraffination):

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The attempts to improve the quality of Brightstock oil, produced by a process developed at Zeitz of using selective solvents or substances with active surfaces, were unsuccessful because of the high viscosity of the oils. After extensive experiments on a laboratory scale, the desired success was attained by using a mixture of catalytic agents in the condensation stage, at the same time simplifying

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the complicated final processing by adding substances with activated surfaces. The oils which have been produced on a semi-technical scale by the new process completely live up to expectations as far as quality is concerned. The final processing with Fuller's earth resulted in good quality compressor and machine oils. The research project was concluded on schedule at the end of plan year 1954.

- g. [] Designation: Individual Hydro-Carbons 25X1
(Einzel-KW):

From the extensive literature concerning the extraction of enriched aromatic extracts or pure aromatics from low-temperature hydrogenation, "Abstreifer", a few processes were chosen which looked promising. By crude and precision distillation it was possible to obtain fractions rich in toluol which can be used successfully instead of pure toluol in the production of "T-ester" by the Zeitz process. On the basis of blueprints delivered, the Heckmannwerk oHG GmbH, Leipzig, is to begin the construction and assembly of a semi-technical installation. The extraction of "Abstreifer" with liquid sulphur dioxide and propane produced 99% enriched aromatic fractions. By extractive distillation with phenol, toluol which was 99% pure was obtained. Attempts to procure background material for a semi-technical installation are being continued. The project will be continued in plan year 1955 (see paragraph 7, e) ~~section~~. The approved plan funds amount to 250,000 DME.

- h. [] Designation: Thiophene: 25X1

As a result of a thorough study of available literature and patents, it was determined that thiophene can be produced in an efficient manner from available cheap raw material, such as butane, sulphur and/or sulphur dioxide. According to the research projects, this process was to be worked out in order to obtain background material for the construction of technical installations. In 1954 the reaction of butane with sulphur dioxide was worked out, optimal conditions were determined, and pure thiophene was produced on a small scale (in kilograms). A small technical apparatus was constructed and set up for the reaction of butane with sulphur because doing it by hand proved very difficult and time consuming. However, the apparatus, as well as the small installation for the sulphur dioxide reaction which is to be set up, could not be tested until 1955 because of technical construction difficulties which were incurred. The project will be continued in 1955 under the designation "ThiopheneProduction". The approved plan funds amount to 200,000 DME. (See paragraph 7, f).

- i. [] Designation: X-Ray Absorption Paste 25X1
(Roentgenabsorptionspaste):

In 1953 an x-ray absorption paste for ferrous metals for the purpose of reducing excessive radiation effects was developed, and in 1954 the paste was put into production. The development and testing of a similar paste for light metals and for non-ferrous metals was carried out in 1954; production of these pastes is to begin in 1955. The project was concluded according to schedule at the end of plan year 1954.

- j. [] Designation: Hydrogenation Catalysts 25X1
(Hydrierkatalysatoren):

Within the framework of this research project, experiments were carried out to determine the relationship between the reactivity of carrier substances, the outer surface structures of contacts, and hydrogenability. Measurements showed that there were qualitative relationships between the phenol content and the aniline point of "Abstreifer"

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fractions on the one hand and the size of the macro surface and/or the radius of the pores of the macropore system on the other hand. The project was concluded according to schedule at the end of 1954.

k. Designation: Removal of Ash from Tar
(Teerentaschung):

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The work begun in 1953 on reducing the high ash content of crude tar was considerably expanded in 1954 for the purpose of attempting to reduce ash content to less than four milligrams per 100 grams. This goal could not be attained, however, and after thorough investigation it was determined that it will be impossible to reduce the ash content of Espenhain tar to the desired amount unless the entire tar processing installation is completely rebuilt. It was determined that the size of the granules of dust in the crude tar and the radii of the macropore system of the filter disk, which amount to about 1.3 microns, affect the purification. Since the radii of the dust particles in Espenhain crude tar are for the most part less than 1.3 microns, about 65% of the dust goes over into the filtrate. This dust can only be removed from the tar by centrifuging, which means that it will be necessary to greatly increase centrifuge capacity. Attempts to purify the tar using ultra sound and sedimentation proved unsuccessful as far as the technical process is concerned. Filtration through a filter disk of magnetized iron powder, a process which may possibly gain in significance, has only been studied in the laboratory and its effectiveness can only be judged after experiments on a technical scale have been made. The research project is to be continued in 1955. The approved plan amounts to 100,000 DME.

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5. According to the approved research and development plan for 1955, a total of seven research projects were undertaken by the research and development office of VEB Hydrierwerk Zeitz during the first quarter of 1955. Of these, six were carried out by the Research and Development Office itself, and one was carried out in conjunction with the Technology Department and the Measurements Department. The list of research projects for 1955 was drawn up at VEB Hydrierwerk Zeitz and the appropriate work circles of the Main Department for Research and Technical Development were informed. After the research projects had been coordinated with the projects of other plants under the Ministry for Heavy Industry, the work circles officially proposed that they be carried out. The Ministry then approved the research projects contained in the plan and established plan funds for 1955 amounting to 1,200,000 DME. During the first quarter of 1955 general costs for the seven research projects amounted to 187,262 DME.
6. The following table lists the seven research projects with their designations, plan numbers, personnel in charge of the projects, and the plan funds for the year 1955:

Designation	Personnel in charge of Project	Plan Funds (thousands of DME)		
		Total	General Costs	Investment Costs
Refining and analysis of lubricating oils	Dr. Kuehnhanss Ing. Roessner	120	105	15
Hydro-Carbon Analysis	Dr. Kuehnhanss	130	120	10
T-ester	D. C. Seumel Dr. Reinhardt Ing. Dietze	300	220	80

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Ring Expansion	Dr. Kuehnhanss	100	90	10	
	Cand. Chem. Illig				
Individual Hydro- Carbons	Ing. Huettig	250	150	100	
Thiophen Production	Dr. Teubel	200	150	50	25X1
Removal of Ash from Tar	DI. Greber	100	90	10	
	D.Ph.Kressner				
	TOTAL:	1,200	925	275	

7. The following is a short description of the individual research projects carried out by the Research and Development Office during the first quarter of 1955:

a. Refining and Analysis of Lubricating Oil:

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General costs for the first quarter of 1955 amounted to 22,400 DME.

The overall work is divided into three individual sections:

- (1) Development of component parts of synthetic oils. Various synthetic oils were produced. In all cases white paraffin was used as a starting component or coupling component (Kupplungskomponente). Thus, chlorinated white paraffin was coupled with aromatics and dehalogenated. Furthermore, white paraffin was subjected to a cracking distillation and the olefin crack distillate thus obtained was polymerized to produce oils. The object of the research is to determine relationships between physical and chemical properties and chemical constitution after the basic components of the oils have been determined.
- (2) Development of a rapid analysis method. First, analyses were made of the spindle oils and machine oils which were produced every day. The triangle method which was developed in 1954 was used to determine the basic components of the oils.
- (3) Refining of lubricating oil. Oil from Herrenleite was subjected to hydrogenation refining. The result was a slightly yellow, strongly fluorescent oil. According to results obtained thus far, this product can be used as white oil for precision mechanics.

b. Hydro-Carbon Analysis:

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General costs for the first quarter of 1955 amounted to 22,600 DME.

During the first quarter of 1955 several special "Abstreifer" from the low-temperature hydrogenation and high-temperature medium-pressure process were precision-distilled, and the component parts were separated out according to the procedures developed by Gooding, Adams and Rall. An attempt will be made to work out a method for quickly separating out hydro-carbon groups in the boiling range of gasoline. Investigation of Espenhain light oil were begun. Since 15 January 1955, the experimental part of the work has been carried out by Dipl. Ing. Seumel (fmu).

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25X1f. Thiophene Production:

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General costs for the 1st quarter of 1955 amounted to 30,200 DME.

During the first quarter of 1955 study of the reaction of butane with sulphur dioxide to form thiophene was continued on a laboratory scale. Contact 6448, which was used for the reaction, was subjected to an endurance test in order to ascertain how often it could be regenerated without loss of activity. One contact has thus far been regenerated 43 times without even removing it from the reaction tube, and the contact is still just as active as it was in the beginning. Regeneration is carried out by roasting off the contact with air under certain conditions. The life span of a contact is important for setting the price of thiophene. On the basis of laboratory experiments the machine-engineering department of the plant was given plans for the construction of a small technical installation for producing thiophene. Furthermore, experiments in producing thiophene homologues from n-Hexane and SO_2 were carried out. However, the efficiency of the process was slight and a mixture of various thiophene homologues was produced.

g. Removal of Ash from Tar:

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General costs for the first quarter of 1955 amounted to 22,900 DME.

The following report for the first quarter was presented by Dipl. Phys. Kressner (fnu). The processing times of spindle oil which had not been made impure and which had a definite viscosity was determined by the use of crude material (Haufwerk); only the distribution of the granules was varied. Using the same crude material with oil of fixed properties it was discovered that processing times varied up to 15%. It further seems probable that crude material with large granules does not have a lower processing time than crude material with finer granules. Calculations were started in order to determine the granule distribution of "poorly filterable" clouded material.

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